

## PCT COOPERATION TREATY

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## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner  
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 Office, PCT  
 2011 South Clark Place Room  
 CP2/5C24  
 Arlington, VA 22202  
 ETATS-UNIS D'AMERIQUE  
 in its capacity as elected Office

Date of mailing (day/month/year) 30 April 2001 (30.04.01)	
International application No. PCT/EP00/07910	Applicant's or agent's file reference P1999S004
International filing date (day/month/year) 11 August 2000 (11.08.00)	Priority date (day/month/year) 17 August 1999 (17.08.99)
Applicant HOLT, David, Gary, Lawton	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:  
 13 March 2001 (13.03.01)

☐ in a notice effecting later election filed with the International Bureau on:  
 \_\_\_\_\_

2. The election ☒ was  
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Jean-Marie McAdams Telephone No.: (41-22) 338.83.38
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(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
22 February 2001 (22.02.2001)

PCT

(10) International Publication Number  
**WO 01/12762 A1**

(51) International Patent Classification<sup>7</sup>: **C10M 163/00** //  
(C10M 163/00, 159:12, 129:42, 129:93)

Lawton [GB/US]; 18 Saddler Drive, Medford, NJ 08055 (US).

(21) International Application Number: **PCT/EP00/07910**

(74) Agents: **DEW, Melvyn, John** et al.; ExxonMobil Chemical Europe Inc., P.O. Box 105, B-1830 Machelen (BE).

(22) International Filing Date: 11 August 2000 (11.08.2000)

(81) Designated States (*national*): CA, JP, SG, US.

(25) Filing Language: English

(84) Designated States (*regional*): European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

(26) Publication Language: English

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9919490.4 17 August 1999 (17.08.1999) GB

**Published:**

- With international search report.
- Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.

(71) Applicant (*for all designated States except US*): **EXXON-MOBIL RESEARCH AND ENGINEERING COMPANY** [US/US]; 1545 Route 22 East, Clinton Township, Annandale, NJ 08801 (US).

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(72) Inventor; and

(75) Inventor/Applicant (*for US only*): **HOLT, David, Gary,**



**WO 01/12762 A1**

(54) Title: CRYSTAL FORMATION INHIBITION IN LUBRICATING COMPOSITIONS

(57) Abstract: Lubricating oil formulations comprising base oil, such sulfur-phosphorous anti-wear/extreme pressure agents and such hindered phenol antioxidants which anti-wear/extreme-pressure agents and hindered phenolic antioxidants are prone to react and form crystals wherein the base oil is characterized as having a saturates content of less than 99 % which base oil is stabilized against the above mentioned crystal formation by the addition of a minor amount of a high molecular weight di- or polycarboxylic acid anhydride, or mixture thereof.

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## REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference  
(if desired) (12 characters maximum)

P1999S004

## Box No. I TITLE OF INVENTION

STABILIZED LUBRICATING FORMULATION AND METHOD

## Box No. II APPLICANT

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

EXXON RESEARCH AND ENGINEERING COMPANY  
Exxon Mobil Corporation  
Downstream Law, Research and Engineering  
1545 Route 22 East  
Annandale, NJ 08801-0900  
USA

☐ This person is also inventor.

Telephone No.

Facsimile No.

Teleprinter No.

State (that is, country) of nationality: US

State (that is, country) of residence: US

This person is applicant  
for the purposes of:☐ all designated  
States☒ all designated States except  
the United States of America☐ the United States  
of America only☐ the States indicated in  
the Supplemental Box

## Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

HOLT, David Gary Lawton  
18 Saddler Drive  
Medford, NJ 08055  
USA

This person is:

☐ applicant only☒ applicant and inventor☐ inventor only (If this check-box  
is marked, do not fill in below.)

State (that is, country) of nationality: UK

State (that is, country) of residence: US

This person is applicant  
for the purposes of:☐ all designated  
States☐ all designated States except  
the United States of America☒ the United States  
of America only☐ the States indicated in  
the Supplemental Box☐ Further applicants and/or (further) inventors are indicated on a continuation sheet.

## Box No. IV AGENT OR COMMON REPRESENTATIVE: OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf  
of the applicant(s) before the competent International Authorities as:

☒ agent☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

DEW, Melvyn John ; MARESCHAL, Anne M. ; VELDHUIZEN,  
Albert Dirk Willem  
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☐ Address for correspondence: Mark this check-box where no agent or common representative has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

**Box No.V DESIGNATION OF STATES**

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked):

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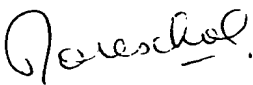
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| <input type="checkbox"/> KZ Kazakhstan                            |   |

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**Precautionary Designation Statement:** In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation (including fees) must reach the receiving Office within the 15-month time limit.)

<b>Box No. VI PRIORITY CLAIM</b>					<input type="checkbox"/> Further priority claims indicated in the Supplemental Box.
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:			
		national application: country	regional application: regional Office	international application: receiving Office	
item (1) 17 AUGUST 1999	9919490.4	UK			
item (2)					
item (3)					
<input type="checkbox"/> The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office) identified above as item(s):					
<i>* Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.</i>					
<b>Box No. VII INTERNATIONAL SEARCHING AUTHORITY</b>					
Choice of International Searching Authority (ISA) (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):		Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority):			
ISA / EP		Date (day/month/year)	Number	Country (or regional Office)	
<b>Box No. VIII CHECK LIST; LANGUAGE OF FILING</b>					
This international application contains the following number of sheets: request : 3 description (excluding sequence listing part) : 15 claims : 3 abstract : 1 drawings : sequence listing part of description : Total number of sheets : 22		This international application is accompanied by the item(s) marked below: 1. <input checked="" type="checkbox"/> fee calculation sheet 2. <input type="checkbox"/> separate signed power of attorney 3. <input checked="" type="checkbox"/> copy of general power of attorney; reference number, if any: 55, 18826 4. <input type="checkbox"/> statement explaining lack of signature 5. <input type="checkbox"/> priority document(s) identified in Box No. VI as item(s): 6. <input type="checkbox"/> translation of international application into (language): 7. <input type="checkbox"/> separate indications concerning deposited microorganism or other biological material 8. <input type="checkbox"/> nucleotide and/or amino acid sequence listing in computer readable form 9. <input type="checkbox"/> other (specify): Acknowledgement of receipt form			
Figure of the drawings which should accompany the abstract:		Language of filing of the international application: EN			
<b>Box No. IX SIGNATURE OF APPLICANT OR AGENT</b>					
Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).					
 Anne Mareschal General Authorization No. 55					

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1. Date of actual receipt of the purported international application:	2. Drawings:
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# PCT

## FEE CALCULATION SHEET Annex to the Request

For receiving Office use only

International application No.

Applicant's or agent's  
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P1999S004

Date stamp of the receiving Office

Applicant

EXXON RESEARCH AND ENGINEERING COMPANY

### CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE . . . . . EUR . . . . . 102 . . . . . ☐ T

2. SEARCH FEE . . . . . EUR . . . . . 945 . . . . . ☐ S

International search to be carried out by EP  
(If two or more International Searching Authorities are competent in relation to the international application, indicate the name of the Authority which is chosen to carry out the international search.)

### 3. INTERNATIONAL FEE

#### Basic Fee

The international application contains 22 sheets.

first 30 sheets . . . . . EUR . . . . . 413 . . . . . ☐ b1

remaining sheets x additional amount = . . . . . ☐ b2

Add amounts entered at b1 and b2 and enter total at B . . . . . 413 . . . . . ☐ B

#### Designation Fees

The international application contains 5 designations.

5 x 95 = . . . . . 475 . . . . . ☐ D

number of designation fees payable (maximum 8) amount of designation fee

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4. FEE FOR PRIORITY DOCUMENT (if applicable) . . . . . ☐ P

5. TOTAL FEES PAYABLE . . . . . EUR . . . . . 1935

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TOTAL

☐ The designation fees are not paid at this time.

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28300171

11 August 2000

*Research*

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1 ALLGEMEINE VOLLMACHT  
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AV Nr. (bei jeder Korrespondenz angeben)  
GA No. (please quote in all correspondence)  
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55 (rev.)

2 Ich (Wir) / I (We) / Je (Nous)

ExxonMobil Research and Engineering Company,  
Florham Park,  
New Jersey,  
United States of America,  
Incorporated in the State of Delaware

3 bevollmächtige(n) hiermit / do hereby authorise / autorise (autorisons) par la présente

SOMERS, Harold Arnold

FLETCHER WATTS, Susan Jane

of: Esso Engineering (Europe) Ltd., Patents and Licences, Mailpoint  
70, Esso House, Ermyn Way, Leatherhead, Surrey KT22 8XE. United  
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DEW, Melvyn John

of: Exxon Chemical Europe Inc., Hermeslaan 2, B-1831, Machelen,  
Belgium.

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to represent me (us) in all proceedings established by the European Patent Convention and to act for me (us) in all patent transactions and to receive  
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à me (nous) représenter pour ce qui concerne toutes mes (nos) affaires de brevet dans toute procédure instituée par la Convention sur le brevet européen  
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Datum / Date 07 January 2000

Unterschrift(en) / Signature(s)

HAROLD ARNOLD SOMERS (- *Quiff*)  
Authorised signatory

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Ich (Wir) / I (We) / Je (Nous)

EXXONMOBIL RESEARCH & ENGINEERING COMPANY  
180 Park Avenue  
Florham Park, NJ 07932  
U.S.A.

bevollmächtigte(n) hiermit / do hereby authorise / autorise (autorisons) par la présente

Anne MARESCHAL,  
having a professional address at  
Exxon Chemical Europe Inc., Law Technology, Hermeslaan 2, B-1831 Machelen, Belgium

mich (uns) in den durch das Europäische Patentübereinkommen geschaffenen Verfahren in allen meinen (unseren) Patentangelegenheiten zu vertreten, alle Handlungen für mich (Uns) vorzunehmen und Zahlungen für mich (Uns) in Empfang zu nehmen.

to represent me (us) in all proceedings established by the European Patent Convention and to act for me (us) in all patent transactions and to receive payments on my (our) behalf.

à me (nous) représenter pour ce qui concerne toutes mes (nos) affaires de brevet dans toute procédure instituée par la Convention sur le brevet européen et, à ce titre, à agir en mon (notre) nom et à recevoir des paiements pour mon (notre) compte.

☒ Die Vollmacht gilt auch für Verfahren nach dem Vertrag über die internationale Zusammenarbeit auf dem Gebiet des Patentrechts.  
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☒ Untervollmacht kann erteilt werden. / Sub-authorisation may be given. / Le pouvoir pourra être délégué.

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Please return the yellow copy, supplemented by the General Authorisation No., to the authorisor.  
Prière de renvoyer la copie jaune au mandant, munie du n° du pouvoir général.

Ort/Place/Lieu Machelen, Belgium

Datum / Date 22 May 2000

Unterschrift(en) / Signature(s)

Melvyn John DEW  
Authorised Signatory

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Le formulaire doit être signé de la propre main du (des) mandant(s) (dans le cas de personnes morales, de la personne ayant qualité pour signer). Veuillez ajouter à la machine, après la signature, le (les) nom(s) du (des) signataire(s) en mentionnant, dans le cas de personnes morales, ses (leurs) fonctions au sein de la société.



**GENERAL AUTHORISATION<sup>1</sup>**

For official use only  
General Authorisation N°

18826

I (We)<sup>2</sup>

EXXON RESEARCH AND ENGINEERING COMPANY, a Corporation duly organized and existing under the laws of the State of Delaware, United States of America

PO Box 390, Florham Park, New Jersey 07932,  
United States of America

do hereby authorise<sup>3</sup>

VELDHUIZEN, Albert Dirk Willem

Exxon Chemical Limited  
Exxon Chemical Technology Centre  
PO Box 1  
Abingdon  
Oxfordshire OX13 6BB  
United Kingdom

☐ (Additional representatives indicated on supplementary sheet).

to represent me (us) in all proceedings established by the European Patent Convention and to act for me (us) in all patent transactions and to receive payments on my (our) behalf.

☒ Substitute authorisation may be given.

Place Abingdon, United Kingdom

Date 26 November 1987

Signature(s)<sup>4</sup>



BAWDEN, Peter Charles - Authorised Signatory  
Chief European Patent Attorney

- Please supplement signature(s) by typewritten name(s) -

# PATENT COOPERATION TREATY

From the RECEIVING OFFICE

# PCT

To:

Dew, Melvyn John  
EXXONMOBIL CHEMICAL EUROPE INC.  
P.O. Box 105  
B-1830 Machelen  
BELGIQUE

## NOTIFICATION OF THE INTERNATIONAL APPLICATION NUMBER AND OF THE INTERNATIONAL FILING DATE

(PCT Rule 20.5(c))

Date of mailing  
(day/month/year)

04 OCT 2000

Applicant's or agent's file reference

P1999S004

### IMPORTANT NOTIFICATION

International application No.

PCT/EP 00/07910

International filing date (day/month/year)

11/08/2000

Priority date (day/month/year)

17/08/1999

Applicant

EXXON RESEARCH AND ENGINEERING COMPANY

Title of the invention

1. The applicant is hereby notified that the international application has been accorded the international application number and the international filing date indicated above.
2. The applicant is further notified that the record copy of the international application was transmitted to the International Bureau on the above date of mailing.

3. ☐ Other:

\* The International Bureau monitors the transmittal of the record copy by the receiving Office and will notify the applicant (with Form PCT/IB/301) of its receipt. Should the record copy not have been received by the expiration of 14 months from the priority date, the International Bureau will notify the applicant (Rule 22.1(c)).

Name and mailing address of the receiving Office



European Patent Office, P.B. 5818 Patentlaan 2  
NL-2280 HV Rijswijk  
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Authorized officer

J. A. J. M. P. G. O. H. E.

## PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF RECEIPT OF  
RECORD COPY

(PCT Rule 24.2(a))

To:

DEW, Melvyn, John  
ExxonMobil Chemical Europe Inc.  
P.O. Box 105  
B-1830 Machelen  
BELGIQUE

Date of mailing (day/month/year) 31 October 2000 (31.10.00)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference P1999S004	International application No. PCT/EP00/07910

The applicant is hereby notified that the International Bureau has received the record copy of the international application as detailed below.

Name(s) of the applicant(s) and State(s) for which they are applicants:

EXXON RESEARCH AND ENGINEERING COMPANY (for all designated States except US)

HOLT, David, Gary, Lawton (for US)

International filing date : 11 August 2000 (11.08.00)

Priority date(s) claimed : 17 August 1999 (17.08.99)

Date of receipt of the record copy  
by the International Bureau : 09 October 2000 (09.10.00)

List of designated Offices :

EP : AT,BE,CH,CY,DE,DK,ES,FI,FR,GB,GR,IE,IT,LU,MC,NL,PT,SE

National : CA,JP,SG,US

## ATTENTION

The applicant should carefully check the data appearing in this Notification. In case of any discrepancy between these data and the indications in the international application, the applicant should immediately inform the International Bureau.

In addition, the applicant's attention is drawn to the information contained in the Annex, relating to:

- ☒ time limits for entry into the national phase
- ☒ confirmation of precautionary designations
- ☒ requirements regarding priority documents

A copy of this Notification is being sent to the receiving Office and to the International Searching Authority.

<p>The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland</p> <p>Facsimile No. (41-22) 740.14.35</p>	<p>Authorized officer:</p> <p>Athina Nickitas-Etienne</p> <p>Telephone No. (41-22) 338.83.38</p>
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K. STORMS

# PATENT COOPERATION TREATY

19 JAN 2001 PCT

## NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and  
Administrative Instructions, Section 422)

From the INTERNATIONAL BUREAU

To:

DEW, Melvyn, John  
ExxonMobil Chemical Europe Inc.  
P.O. Box 105  
B-1830 Machelen  
BELGIQUE

Date of mailing (day/month/year) 09 January 2001 (09.01.01)	<b>IMPORTANT NOTIFICATION</b>
Applicant's or agent's file reference P1999S004	
International application No. PCT/EP00/07910	International filing date (day/month/year) 11 August 2000 (11.08.00)

1. The following indications appeared on record concerning:

☒ the applicant ☐ the inventor ☐ the agent ☐ the common representative

Name and Address

EXXON RESEARCH AND ENGINEERING  
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Exxon Mobil Corporation  
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1545 Route 22 East  
Annandale, NJ 08801-0900  
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State of Nationality

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State of Residence

US

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2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

☐ the person ☒ the name ☒ the address ☐ the nationality ☐ the residence

Name and Address

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State of Residence

US

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Teleprinter No.

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

☒ the receiving Office ☐ the designated Offices concerned  
☒ the International Searching Authority ☐ the elected Offices concerned  
☐ the International Preliminary Examining Authority ☐ other:

The International Bureau of WIPO  
34, chemin des Colombettes  
1211 Geneva 20, Switzerland

Facsimile No.: (41-22) 740.14.35

Authorized officer

Athina Nickitas-Etienne

Telephone No.: (41-22) 338.83.38

*Handwritten:* 26.01.01

## PATENT COOPERATION TREATY

1) To KS/NC FOR DE

Kaake

2 MAR. 2001

PCT

From the INTERNATIONAL BUREAU

NOTICE INFORMING THE APPLICANT OF THE  
COMMUNICATION OF THE INTERNATIONAL  
APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

To:

DEW, Melvyn, John  
ExxonMobil Chemical Europe Inc.  
P.O. Box 105  
B-1830 Machelen  
BELGIQUE

Date of mailing (day/month/year)

22 February 2001 (22.02.01)

Applicant's or agent's file reference

P1999S004

## IMPORTANT NOTICE

International application No.

PCT/EP00/07910

International filing date (day/month/year)

11 August 2000 (11.08.00)

Priority date (day/month/year)

17 August 1999 (17.08.99)

Applicant

EXXONMOBIL RESEARCH AND ENGINEERING COMPANY et al

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:

US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

CA,EP,JP,SG

The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on 22 February 2001 (22.02.01) under No. WO 01/12762

## REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a **demand for international preliminary examination** must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

## REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the **national phase**, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

The International Bureau of WIPO  
34, chemin des Colombettes  
1211 Geneva 20, Switzerland

Facsimile No. (41-22) 740.14.35

Authorized officer

J. Zahra

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Continuation of Form PCT/IB/308

NOTICE INFORMING THE APPLICANT OF THE COMMUNICATION OF  
THE INTERNATIONAL APPLICATION TO THE DESIGNATED OFFICES

<b>Date of mailing (day/month/year)</b> 22 February 2001 (22.02.01)	<b>IMPORTANT NOTICE</b>
<b>Applicant's or agent's file reference</b> P1999S004	<b>International application No.</b> PCT/EP00/07910
<p>The applicant is hereby notified that, at the time of establishment of this Notice, the time limit under Rule 46.1 for making amendments under Article 19 has not yet expired and the International Bureau had received neither such amendments nor a declaration that the applicant does not wish to make amendments.</p>	

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
22 February 2001 (22.02.2001)

PCT

(10) International Publication Number  
**WO 01/12762 A1**

(51) International Patent Classification<sup>7</sup>: **C10M 163/00 //**  
(C10M 163/00, 159:12, 129:42, 129:93)

**Lawton** [GB/US]; 18 Saddler Drive, Medford, NJ 08055 (US).

(21) International Application Number: **PCT/EP00/07910**

(74) Agents: **DEW, Melvyn, John** et al.; ExxonMobil Chemical Europe Inc., P.O. Box 105, B-1830 Machelen (BE).

(22) International Filing Date: **11 August 2000 (11.08.2000)**

(81) Designated States (*national*): CA, JP, SG, US.

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**Published:**

- With international search report.
- Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.

(71) Applicant (*for all designated States except US*): **EXXON-MOBIL RESEARCH AND ENGINEERING COMPANY** [US/US]; 1545 Route 22 East, Clinton Township, Annandale, NJ 08801 (US).

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(72) Inventor; and

(75) Inventor/Applicant (*for US only*): **HOLT, David, Gary,**



**WO 01/12762 A1**

(54) Title: **CRYSTAL FORMATION INHIBITION IN LUBRICATING COMPOSITIONS**

(57) Abstract: Lubricating oil formulations comprising base oil, such sulfur-phosphorous anti-wear/extreme pressure agents and such hindered phenol antioxidants which anti-wear/extreme-pressure agents and hindered phenolic antioxidants are prone to react and form crystals wherein the base oil is characterized as having a saturates content of less than 99 % which base oil is stabilized against the above mentioned crystal formation by the addition of a minor amount of a high molecular weight di- or polycarboxylic acid anhydride, or mixture thereof.

**CRYSTAL FORMATION INHIBITION IN LUBRICATING COMPOSITIONS**

5

This invention relates to lubricating oil based on base stocks having  
10 less than 99 wt% saturates content and containing one or more sulfur-  
phosphorus containing anti-wear/extreme pressure additives and one or more  
hindered phenol anti oxidants which combination are prone to crystal formation,  
wherein the formation of crystals is reduced or eliminated by the use of a  
crystallization suppressant.

15

Lubricating oils containing various antioxidants or esters or fatty  
acid amides or sulfur-phosphorus additives in combination with phenols are  
known in the literature.

20

U.S. Patent 5,167,844 is directed to a formulation comprising a base  
oil, at least one sulfur phosphorus containing compound, at least one amine and  
at least one hindered phenol.

25

JP 07034078 is directed to a hydraulic oil comprising mineral oil  
with an aromatic content of up to 1.5 wt% and a phenolic and aminic anti-  
oxidant, an alkenyl succinic acid imide rust inhibitor and a phosphoric acid type  
anti wear agent.

30

U.S. Patent 5,580,483 is directed for lubricating a refrigeration  
system compressor using a break-in lubricating oil which is an ester type oil.  
Additionally an adipate, phthalate, azelate, sebacate, trimellitate can also be  
present as well as tri hydrocarbonyl phosphate, corrosion inhibitors such as alkali  
and/or alkaline earth metal sulfonate, antioxidants such as aminic or phenolic  
antioxidants and metal deactivators such as triazoles.



5

WO 97/14776 is directed to hydraulic oils comprising base oils combined with an amine antioxidant, a phenolic antioxidant, a phosphate ester and a fatty acid amide and/or polyhydric alcohol ester.

10

U.S. Patent 5,773,393 is directed to a composition comprising at least 70 wt% oil of lubricating viscosity and an amount effective to inhibit metal corrosion of a soluble additive comprising (a) at least one amide compound of a mono- or polycarboxylic acid or reactive derivative thereof and (b) at least 0.5 equivalents of at least one primary or secondary amine per mole of amide provided that when (a) is an amide of a dicarboxylic acid and the amine is an alkanol amine the mixture contains more than 0.5 equivalent of the amine (b) per equivalent of the amide.

15

The present invention is directed to a lubricating oil formulation having a reduced potential for the formation of crystals comprising a major amount of a lubricating oil base stock having less than about 99 wt% saturates content, preferably less than about 98 wt% saturates content, and a minor amount of additives comprising a mixture of sulfur-phosphorus containing anti-wear/extreme pressure additive, hindered phenol antioxidant and one or more high molecular weight di-, or polycarboxylic acid, anhydride or mixture thereof such as polyolefin succinic acid/anhydride, and to a method for reducing crystal formation in lubricating oil formulations comprising base oil having less than about 99 wt% saturates content, preferably less than about 98 wt% saturates content, and containing sulfur phosphorus anti-wear/extreme pressure additive and hindered phenolic anti-oxidant wherein the crystals are attributed to the interaction between the sulfur phosphorus containing anti-wear/extreme pressure agent and the hindered phenol by adding to said lubricating oil a minor effective amount of one or more high molecular weight di- or polycarboxylic acid or

20

25

30

5 anhydride such as polyolefin succinic acid/poly olefin succinic anhydride and/or mixtures thereof.

The lubricating base oil is any oil of lubricating oil viscosity having less than about 99 wt% saturates content, preferably less than about 98 wt%  
10 saturates content.

Lubricating oils meeting this criterion are any natural mineral or petroleum based lubricating oils derived from crude oil, tar sands, shale oil, etc., such that they contain a quantity of unsaturation resulting in a saturates content  
15 of less than of 99%, or a mixture of natural mineral or petroleum based lubricating oils in combination with a base oil or oils having a saturates content of greater than 99 wt%, e.g. hydrocarbon oils such as white oils and/or severely hydrotreated, hydrocracked mineral oils, or synthetic oils such as poly alpha olefins, esters, isomerized wax or isomerized Fischer-Tropsch wax, the  
20 combination or mixture of such oils being characterized as having less than about 99 wt% saturates. Saturates content, for the purposes of this specification, is a measure of the absence of aromatic species, and was determined by high pressure liquid chromatography (HPLC) according to method IP 368, except where otherwise expressly indicated.

25

The lubricating oil base stocks useful in the present invention have the typical lubricating oil viscosity, usually possessing kinematic viscosities in the range of about 1.5 to 500 mm<sup>2</sup>/s at 100°C, preferably 5 to 120 mm<sup>2</sup>/s at 100°C.

30

Mineral or petroleum based lubricating oil base stocks can be derived from paraffinic, naphthenic and mixed base crudes. Conventional refinery techniques include distillation, solvent and/or catalytic dewaxing,

5 solvent extraction, hydrofinishing, hydrocracking, vis breaking, deasphalting, etc.

Synthetic lubricating oils that can be used include esters of di- and tri-basic acids, reacted with linear or branched aliphatic alcohols such as C<sub>6</sub>-C<sub>15</sub> alcohols, such as di-2-ethylhexyl sebacate, phthalic ester esters of glycols such as C<sub>13</sub> oxo acid diester or tetraethylene glycol, or complex esters such as one formed from 1 mole of sebacic acid and 2 moles of tetraethylene glycol and 2 moles of 2-ethylhexanoic acid. Other synthetic oils that can be used include synthetic hydrocarbons such as alkyl benzenes, e.g., alkylate bottoms from the alkylation of benzene with tetrapropylene, or the copolymers of ethylene and propylene; silicone oils, e.g., ethyl phenyl polysiloxanes, methyl polysiloxanes, etc.; polyglycol oils, e.g., those obtained by condensing butyl alcohol with propylene oxide; carbonate esters, e.g., the product of reacting C<sub>6</sub> oxo alcohol with ethyl carbonate to form a half ester followed by reaction of the latter with tetraethylene glycol, etc. Other suitable synthetic oils include the polyphenyl ethers, e.g., those having from about 3 to 7 ether linkages and about 4 to 8 phenyl groups.

Other suitable oils are the polyol ester oils made by reacting an aliphatic polyol with carboxylic acid. Aliphatic polyols contain from 4 to 15 carbon atoms and has from 2 to 8 esterifiable hydroxyl groups. Examples of polyols are trimethylolpropane, pentaerythritol, dipentaerythritol, neopentyl glycol, tripentaerythritol and mixtures thereof. The carboxylic acid reactant is selected from aliphatic monocarboxylic acid or mixtures of aliphatic mono carboxylic acids or mixtures of aliphatic mono- and di-carboxylic acids. The carboxylic acids contain 4 to 12 carbons and include straight and branched chain carboxylic acids.

- 5 -

5 Included in the group of synthetic oils are those recovered from tar sands, shale oil, light hydrocarbons produced via, for example, the Fischer-Tropsch process for converting synthesis gas (CO and hydrogen) into hydrocarbons, wax isomerate oils produced by the catalytic hydroisomerization of natural petroleum waxes (i.e., slack wax) or synthetic waxes (i.e., Fischer-Tropsch waxes) or mixtures of such waxes. See USP 5,059,299 and USP 10 5,158,671 for description of wax isomerization and the oils produced thereby. Other synthetic oils include the polyolefins such as polybutene, polyisobutenes and especially the polyalphaolefins, i.e., fluids formed by the oligomerization of at least one 1-alkane hydrocarbon having from 6 to 20 carbons, preferable 8 to 15 16 carbons, more preferably 8 to 12 carbons.

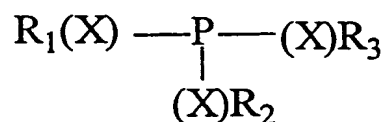
Regardless of the source of the oil, for the purposes of the present invention, the lube oil base stock, be it a single oil or a mixture of oils, is characterized as having a saturates content of less than about 99%, preferably 20 less than 98 wt%.

Sulfur-phosphorus containing anti-wear/extreme pressure additives are well known in the industry, and are materials containing both sulfur and phosphorus in the same molecule. For the purpose of the present specification, 25 and appended claims sulfur-phosphorus containing anti wear, extreme pressure additives are those which react with hindered phenols to produce crystals. Those skilled in the formulation art can readily determine without expenditure of inventive effort, whether a particular sulfur-phosphorus containing anti-wear/extreme pressure agent reacts with hindered phenol anti-oxidant to produce 30 crystals. If it does not, it is not within the scope of this invention. Any sulfur-phosphorus containing anti-wear/extreme pressure agent which is found to react with hindered phenol antioxidant to produce crystals in the subject base oil is within this invention and formalities containing such agents and phenolic

5 antioxidants will be beneficially affected is evidenced by reduction on  
 elimination of crystal formation by the addition of the high molecular weigh di-  
 or poly carboxylic acid, anhydride or mixture thereof, as shown below, provided  
 such carboxylic acid, anhydride or mixture thereof is used in an amount of at  
 least about 0.0013 wt% for each 1 ppm phosphorus attributable to the sulfur-  
 10 phosphorus containing anti-wear/extreme pressure agent.

Sulfur-phosphorus anti-wear/extreme pressure additives which  
 interact with hindered phenols to produce crystals are exemplified by, but not  
 limited to, materials of the type:

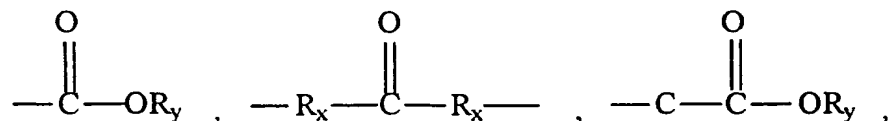
15



wherein  $R_1$ ,  $R_2$  and  $R_3$  are independently hydrogen or hydrocarbyl provided at  
 least one is hydrocarbyl so as to render the material oil soluble and X is sulfur.

20

The hydrocarbyl groups preferably contain from 1 to 40 carbons and  
 are aromatic and/or aliphatic groups and include aryl alkyl and alkaryl and  
 aralkyl and heteroatom substituted aromatic and aliphatic group, the heteroatom  
 substituents being sulfur, nitrogen or oxygen substituted as such into the  
 25 hydrocarbon skeleton or as sulfur, oxygen or nitrogen containing moiety, e.g.,  
 $-OR_y$ ,  $-SH$ ,  $-SO_2H$ ,  $-N(R_y)_2$ ,  $-C-R_xOR_y$ ,

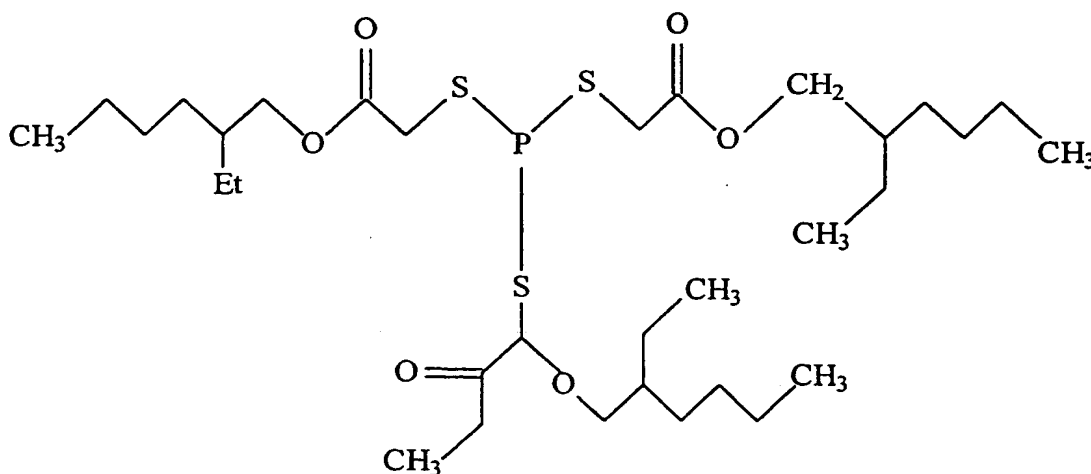


- 7 -

5 etc., and mixtures thereof substituted onto or into the hydrocarbon backbone, wherein  $R_X$  is  $C_1$ - $C_{20}$  hydrocarbyl or hydrocarbylene group and  $R_Y$  is hydrogen or a  $C_1$ - $C_{20}$  hydrocarbyl or hydrocarbylene.

Such sulfur-organo phosphorus containing anti-wear/extreme  
10 pressure agent is typically used at a concentration sufficient to provide of from about 2 ppm to 320 ppm phosphorus, preferably 40 ppm to 200 ppm phosphorus, most preferably about 80 ppm to 130 ppm phosphorus.

An example of a sulfur phosphorus anti-wear/extreme pressure  
15 additive which has been found to react with hindered phenols to form crystals is a material is 2-ethylhexyl 10-ethyl-4-[[2-[(2 ethylhexyl)-oxyl]-2-oxoethyl] thio]-7-oxo-8-oxa-3,5-dithia-4-phospha tetradecanoate, CAS # 83547-95-9. Based on the name and the CAS number, it is believed this material has the following structure:

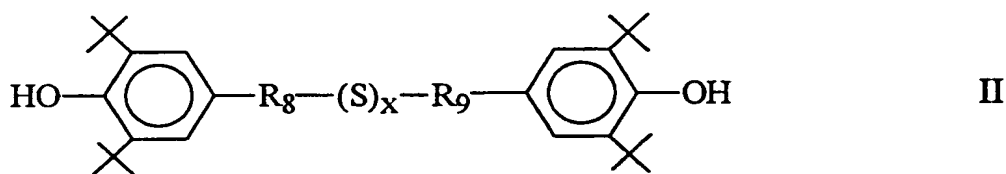


20

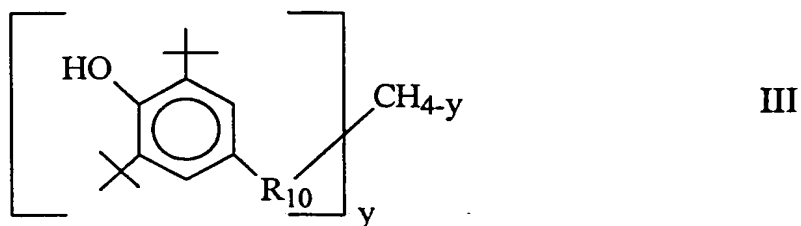
It must be noted that for the purposes of the present invention metal dihydrocarbyldithiophosphate (metal DDP) or ashless DDP do not fall within the above definition of sulfur-phosphorus containing anti-wear/extreme pressure

additive because it has been found that they do not form crystals when combined with hindered phenols in base oils.

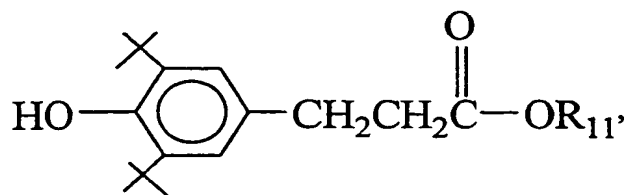
Hindered phenolic anti oxidants are also well known in the industry. Such materials include by way of example and not limitation 2,6-di-t-butyl phenol, 2,6-di-t-butyl alkylated phenol where the alkyl substituent is hydrocarbyl and contains between 1 and 20 carbon atoms, such as 2,6-di-t-butyl-4-methyl phenol, 2,6-di-t-butyl-4-ethyl phenol, etc., or 2,6-di-t-butyl-4-alkoxy phenol where the alkoxy substituent contains between 1 and 20 carbons such as 2,6-di-t-butyl-4-methoxyphenol; materials of the formula



where X is zero to 5, R<sub>8</sub> and R<sub>9</sub> are the same or different and are C<sub>1</sub>-C<sub>20</sub> hydrocarbyl which may contain oxygen or sulfur or be substituted with oxygen or sulfur containing groups; and materials of the formula



where y is 1 to 4 and R<sub>10</sub> is a C<sub>1</sub> to C<sub>20</sub> hydrocarbyl which may contain oxygen sulfur or nitrogen or be substituted with oxygen, sulfur or nitrogen containing groups such as 2,6 di tert butyl α dimethylamino P-cresol,



IV

wherein it is believed R<sub>11</sub> is C<sub>8</sub>C<sub>17</sub> (CAS # 125643-61-0), and mixtures of such phenolic type antioxidants.

Preferably the phenolic anti-oxidant contains an ester group, such as in formula IV above.

Phenolic type anti oxidants are typically used at a concentration of from about 0.01 to 2.0 wt%, preferably about 0.1 to 1.0 wt%, most preferably about 0.3 to 0.5 wt%, based on active ingredient.

In order to prevent or at least minimize the formation of crystals in lubricating oils based on base stock having less than 99% saturates preferably less than 98 wt% saturates and containing a mixture of sulfur-organo phosphorus anti-wear/extreme pressure additive and phenolic anti-oxidant, wherein the sulfur phosphorus containing anti-wear/extreme pressure agent interacts with the hindered phenol to produce crystals a minor, crystal preventing effective amount of a high molecular weight carboxylic acid, anhydride or mixture thereof is added to the lubricating oil formulation.

The carboxylic acid or anhydride can be any high molecular weight acid such as di- or polycarboxylic acid, anhydride or mixture thereof of molecular weight of about 300-5000. Such acids, anhydrides or mixtures thereof include polyhydrocarbylene substituted di- or polycarboxylic acids or anhydrides wherein the poly hydrocarbylene group has a molecular weight in the range 300 to 5000, preferably 750 to 2000, most preferably 900 to 1000 (e.g.,



5 polyisobutylene) and wherein the carboxylic group is, e.g., succinic or maleic acid, anhydride or mixture thereof.

Poly hydrocarbylenes are homopolymer or interpolymers of polymerizable olefin group containing monomers having from 2 to 16 carbons.  
10 Interpolymers are those made using two or more different olefinic groups containing monomer including monomer such as styrenes. Poly hydrocarbylene homo and interpolymers are well known in the literature and to those skilled in the art and need not be further described herein.

15 Preferably the carboxylic acid or anhydride or mixture thereof used is polyalkylene succinic or maleic acid, anhydride, or mixtures thereof, most preferably polyisobutylene (PIB) succinic acid, anhydride or mixtures thereof wherein the PIB group has a molecular weight of about 900 to 1000.

20 Such high molecular weight carboxylic acids, anhydrides are employed in an amount in the range of about 0.0026 to 0.8 wt%, preferably about .08 to 0.4 wt%, most preferably about 0.12 to 0.24 wt%, based on active ingredients.

25 In general, at least 0.0013 wt% of high molecule weight carboxylic acid, anhydride or mixture thereof is used for each 1 ppm phosphorous from the sulfur-organo phosphorus anti-wear/extreme pressure agent.

## EXAMPLES

30

### Example 1

This example (Table 1) is presented to show that, in a base stock having a saturates content of less than 99 wt%, the combination of a sulfur-

- 5 phosphorous anti-wear/extreme pressure agent with a hindered phenol results in crystal formation while the combination of a sulfur free phosphate extreme pressure agent and hindered phenol does not result in crystal formation.

TABLE 1

					Crystals at 3 months
Base oil (1)	+	.55 wt% sulfur-phosphorus extreme pressure agent (2)	+	0.4 wt% hindered phenol (3)	yes
Base oil (1)	+	.4 wt% hindered phenol	+	.55 wt% sulfur free phosphate EP agent (4)	no

10

(1) solvent refined base oil, about 88% saturates 150 SN oil

(2) sulfur phosphorus extreme pressure agent CAS #83547-95-9 which is 60% sulfur-phosphorus component active ingredient (also contained C<sub>4</sub>-C<sub>8</sub> diphenyl amine as balance of additive)

15 (3) 100% active ingredient, CAS # 125643-61-0

(4) 100% active ingredient, isopropylated triaryl phosphate

The resulting lubricant had a phosphorus content of 120ppm by weight, measured according to standard test ASTM D5185-97, attributable to the sulphur-phosphorus extreme pressure agent (which was the sole phosphorus-containing component contained in the lubricant formulation)

20

Example 2

This example (Table 2) is presented to show that crystal formation is  
 25 eliminated in formulations normally exhibiting crystal formation by the addition of high molecular weight anhydride but that crystal formation is not eliminated by the addition of high molecular weight anhydride-poly amine dispersant, or by the addition of esters. All formulations tested in this example further contained typical pour point depressants, anti-rust agent and an amino para cresol  
 30 antioxidant.

TABLE 2

Base oil (1)	+ EP agent (2)	+ .55 wt% sulfur-phosphorus	+ 0.4 wt% hindered phenol (3)	+	Crystals at 3 months
(“)	(“)	(“)	(“)	+ PIBSA + PAM (4)	yes
(“)	(“)	(“)	(“)	+ Esters (5)	yes
(“)	(“)	(“)	(“)	+ PIBSA (6)	yes
(“)	(“)	(“)	(“)	+ PIBSA (7)	no

(1) Base oil, a 50/50 mixture of 150 N (88% saturates) and 400 N (about 78% saturates).

(2) See Table 1.

(3) See Table 1.

(4) PIBSA-PAM was tested at concentration of from 0.05 to .4 wt% and at all concentrations used crystals formed within the three month time period of the test.

(5) Esters tested were di iso nonyl phthalate at 0.05 to 4 wt%; di iso-tridecyl adipate at .1 to .5 wt%; C<sub>6</sub> and C<sub>13</sub> phthalate at .5 wt%. None were effective at eliminating crystal formation during the three month time period of the test.

(6) PIBSA is polyisobutylene succinic anhydride, having a polyisobutylene molecular weight of 950. When used at .04 wt% and .08 wt% active ingredient level, it did not eliminate crystal formation.

(7) PIBSA (of note 6) at .16 wt% and .32 wt% active ingredient level eliminated crystal formation.

5

Example 3

6                   This example (Table 3) is presented to show the effect of base stock  
saturation on the suppression of crystal formation when using PIBSA in  
10 combination with sulfur phosphorus extreme pressure agent and hindered  
phenol.

TABLE 3

		+	0.55 wt% sulfur phosphorus extreme pressure agent (2)	+	0.4 wt% hindered phenol (3)	+	0.16 wt% PIBSA (4)	Crystals at 3 months
Base oil (1)		+						no
Base oil (5)			(“)		(“)		(“)	no
Base oil (6)			(“)		(“)		(“)	no
Base oil (7)			(“)		(“)		(“)	no
Base oil (8)			(“)		(“)		(“)	cloudy
Base oil (9)			(“)		(“)		(“)	cloudy
Base oil (10)			(“)		(“)		(“)	cloudy
Base oil (10)			(“)		(“)		PIBSA @ .8% AI	yes

(1) See Table 1.

(2) See Table 1.

(3) See Table 1.

10 (4) PIBSA is polyisobutylene succinic anhydride, polyisobutylene molecular weight 950.

(5) 150 N, about 80% saturates.

(6) 150N FDA C grade white oil about 80% saturate (by clay-gel analysis - ASTM D 2007).

(7) Hydrocracked 90 N, about 92% saturates.

(8) 150 N FDA A grade white oil, 100% saturates

15 (9) Hydrocracked 150 N, about 99.9% saturates.

(10) PAO-6, 100% saturates.

5

From this it is seen that in base oils having less than 99% saturates and containing mixtures of sulfur-phosphorus extreme pressure agent and hindered phenol, which are prone to crystal formation, crystal formation is suppressed on adding PIBSA where as in base oils of essentially 100% saturates content even addition of PIBSA failed to prevent crystal formation and even increasing PIBSA concentrate to 0.8 wt% (active ingredient) did not prevent crystal formation.

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5 CLAIMS :

1. A lubricating oil of reduced crystal formation potential attributable to the interaction of sulfur phosphorus containing anti-wear/extreme pressure agents and hindered phenolic antioxidants comprising a major amount of a base oil of lubricating viscosity and having less than about 99 wt% saturates content, and a minor amount of additive comprising a sulfur-phosphorus containing anti-wear/extreme pressure additive, a hindered phenol antioxidant and a high molecular weight di- or poly- carboxylic acid, anhydride or mixture thereof provided at least 0.0013 wt% high molecular weight carboxylic acid, anhydride or mixture thereof is present for each 1 ppm phosphorus attributable to the sulfur phosphorus containing anti-wear/extreme pressure agent.

2. The lubricating oil of claim 1 wherein the sulfur-phosphorus anti-wear/extreme pressure agent is in an amount sufficient to provide about 2 ppm to 320 ppm phosphorus, the hindered phenol antioxidant is at a concentration of from about 0.01 to 2.0 wt% based on active ingredient and the high molecular weight di- or poly-carboxylic acid is at a concentration of in the range of about 0.0026 to 0.8 wt% based on active ingredient.

3. The lubricating oil of claim 1 or 2 wherein the sulfur-phosphorus containing anti-wear/extreme pressure agent is in an amount sufficient to provide from 40 ppm to 200 ppm phosphorus.

4. The lubricating oil of claim 1 or 2 wherein the sulfur-phosphorus containing anti-wear/extreme pressure agent is in an amount sufficient to provide from 80 ppm to 130 ppm phosphorus.

5           5. The lubricating oil of any preceding claim wherein the hindered phenol is at a concentration of about 0.1 to 1.0 wt% based on active ingredient.

6. The lubricating oil of claim 2, 3 or 4 wherein the hindered phenol is at a concentration of about 0.3 to 0.5 wt% based on active ingredient.

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7. The lubricating oil of claim 2, 3 or 4 wherein the high molecular weight di- or poly-carboxylic acid, anhydride or mixture thereof is at a concentration of about 0.08 to 0.4 wt% based on active ingredient.

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8. The lubricating oil of any preceding claim wherein the high molecular weight di- or poly-carboxylic acid anhydride or mixture thereof is at a concentration of about 0.12 to 0.24 wt% based on active ingredient.

9. The lubricating oil of any preceding claim wherein the high molecular weight di- or poly-carboxylic acid, anhydride mixture thereof is a polyhydrocarbylene substituted di- or poly-carboxylic acid, anhydride or mixture thereof wherein the polyhydrocarbylene group has a molecular weight in the range 300 to 5,000.

25

10. A method for reducing crystal formation in lubricating oil containing a mixture of sulfur phosphorus anti-wear/extreme pressure agent and hindered phenols antioxidant wherein the sulfur-phosphorus anti-wear/extreme pressure agent interacts with the phenolic antioxidant to produce crystals, such method comprising adding to a major amount of a base oil of lubricating viscosity having a saturates content of less than 99 wt%, a minor amount of additives comprising a sulfur-phosphorus containing anti-wear/extreme pressure agent a hindered phenol antioxidant and a high molecular weight di- or poly-carboxylic acid, anhydride or mixture thereof provided at least 0.0013 wt% of

30



- 5 the high molecular weight di- or poly-carboxylic acid, anhydride or mixture thereof is used for each 1 ppm phosphorus attributable to the sulfur-phosphorus containing anti-wear/extreme pressure agent.

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP 00/07910

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C10M163/00 //(C10M163/00,159:12,129:42,129:93)

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C10M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EP0-Internal

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 458 495 A (PANZER JEROME ET AL) 29 July 1969 (1969-07-29) column 1, line 33 -column 1, line 41 column 2, line 53 -column 3, line 29 example 1 column 6, line 63 -column 8, line 29 ---	1-10
A	US 2 766 207 A (JOHN PATRICK MCDERMOTT ET AL) 9 October 1956 (1956-10-09) the whole document -----	1-10

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*G\* document member of the same patent family

Date of the actual completion of the international search

30 January 2001

Date of mailing of the international search report

05/02/2001

Name and mailing address of the ISA

European Patent Office, P.B. 5618 Patentlaan 2  
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Fax: (+31-70) 340-3016

Authorized officer

Perakis, N

# INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/EP 00/07910

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 3458495 A	29-07-1969	NONE	
US 2766207 A	09-10-1956	NONE	

# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference <b>P1999S004</b>	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. <b>PCT/EP00/07910</b>	International filing date (day/month/year) <b>11/08/2000</b>	Priority date (day/month/year) <b>17/08/1999</b>
International Patent Classification (IPC) or national classification and IPC <b>C10M163/00</b>		
Applicant <b>EXXONMOBIL RESEARCH AND ENGINEERING COMPANY et al.</b>		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
  
2. This REPORT consists of a total of 4 sheets, including this cover sheet.
 

☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I    ☒ Basis of the report
- II   ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V    ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand  <b>13/03/2001</b>	Date of completion of this report  <b>05.04.2001</b>
Name and mailing address of the international preliminary examining authority:  <div style="display: flex; align-items: center;"> <div>             European Patent Office              D-80298 Munich              Tel. +49 89 2399 - 0 Tx: 523656 epmu d              Fax: +49 89 2399 - 4465           </div> </div>	Authorized officer  <b>Perakis, N</b>  Telephone No. +49 89 2399 8355



**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/EP00/07910

**I. Basis of the report**

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

**Description, pages:**

1-15 as originally filed

**Claims, No.:**

1-10 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/EP00/07910

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Yes: Claims 1-10
	No: Claims
Inventive step (IS)	Yes: Claims 1-10
	No: Claims
Industrial applicability (IA)	Yes: Claims 1-10
	No: Claims

2. Citations and explanations  
**see separate sheet**

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

---

International application No. PCT/EP00/07910

**Re Item V**

**Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

The present international application concerns a lubricating composition comprising a base oil and various additives. The base oil is one containing less than 99 wt % of saturates. The additives are a specific mixture of a S/P antiwear additive and a hindered phenol antioxidant additive which are so selected that they react. The composition also contains an additional additive which is a di/poly acid anhydride. This additive stabilizes the base oil in view of the crystal formed by the reaction product of the two previous additives.

The subject-matter claim is novel over the state of the art. D1: US-A-3458495 (cf. passages cited in the International Search Report) discloses the reaction between a P/S antiwear additive and a hindered phenol. It does not however disclose the specific combination of the previous additives with a di/poly acid anhydride.

The technical problem this combination solves is the stabilisation of the base oils with a saturates content of less than 99 wt%. The solution of the set technical problem does not seem to be obvious since it does not derive from the state of the art and does not seem to belong to the general technical knowledge of the person skilled in the art.

# PATENT COOPERATION TREATY

# PCT

## INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference <b>P1999S004</b>	<b>FOR FURTHER ACTION</b> see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. <b>PCT/EP 00/ 07910</b>	International filing date (day/month/year) <b>11/08/2000</b>	(Earliest) Priority Date (day/month/year) <b>17/08/1999</b>
Applicant  <b>EXXON RESEARCH AND ENGINEERING COMPANY et al.</b>		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

**1. Basis of the report**

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of invention is lacking** (see Box II).

4. With regard to the **title**,

☐ the text is approved as submitted by the applicant.

☒ the text has been established by this Authority to read as follows:

**CRYSTAL FORMATION INHIBITION IN LUBRICATING COMPOSITIONS**

5. With regard to the **abstract**,

☐ the text is approved as submitted by the applicant.

☒ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☒ None of the figures.



**B x III TEXT OF THE ABSTRACT (Continuation of item 5 of the first sheet)**

Lubricating oil formulations comprising a base-oil, such a sulfur-phosphorous anti-wear/extreme pressure agents and such hindered phenol antioxidants which anti-wear/extreme-pressure agents and hindered phenolic antioxidants are prone to react and form crystals wherein the base oil is characterized as having a saturates content of less than 99% which base oil is stabilized against the above mentioned crystal formation by the addition of a minor amount of a high molecular weight di- or polycarboxylic acid anhydride, or mixture thereof.

# INTERNATIONAL SEARCH REPORT

International Application No

EP 00/07910

**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC 7 C10M163/00 //(C10M163/00, 159:12, 129:42, 129:93)

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C10M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 458 495 A (PANZER JEROME ET AL) 29 July 1969 (1969-07-29) column 1, line 33 -column 1, line 41 column 2, line 53 -column 3, line 29 example 1 column 6, line 63 -column 8, line 29 ---	1-10
A	US 2 766 207 A (JOHN PATRICK MCDERMOTT ET AL) 9 October 1956 (1956-10-09) the whole document -----	1-10

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

\* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*&\* document member of the same patent family

Date of the actual completion of the international search

30 January 2001

Date of mailing of the international search report

05/02/2001

Name and mailing address of the ISA

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Authorized officer

Perakis, N

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

EP 00/07910

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 3458495	A	29-07-1969	NONE	
US 2766207	A	09-10-1956	NONE	